MICROSTRUCTURES AND PROPERTIES OF COCRMO ALLOY BY METAL INJECTION MOULDING PROCESS

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SUPERVISOR'S DECLARATION

We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Master of Science.

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I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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Thesis submitted in fulfillment of the requirements
for the award of the degree of
Master of Science

Faculty of Mechanical and Manufacturing Engineering

UNIVERSITI MALAYSIA PAHANG

MAY 2019

ACKNOWLEDGEMENTS

Syukur Alhamdulillah, the first word that resembles everything for His precious given. With His blessing, after all, my research work had finally come to an end within the given timeframe. I would like to extend my heartfelt gratitude to all that have been contributed in this project especially to my supervisor, PM Dr Wan Sharuzi Wan Harun, and my co-supervisor, Dr Nur Zalikha Khalil for their encouragement, guidance and advice during one and half semesters to completely finish this research work.

I also would like to express my particular thanks to Universiti Malaysia Pahang (UMP) for giving me the opportunity and support to carry out my research successfully. My sincere appreciation also goes to Mr Zukarnain, technician staff of Mechanical Engineering Faculty for his help and cooperation. I greatly acknowledge all Green Research for Advanced Materials Laboratory (GRAMSlab)'s teams for their helping hand and lessons during my research activities. A significant contribution and hard working from them to cooperate with me while conducting and completing this thesis was great indeed.

Continued prayers and support from my husband, Mohamad Iamie Bin Rosli, I'm really thankful, thank you for being a great companion and truly understand each of my hard day. I also would like to express sincere appreciation to my beloved family especially my beloved parents Dandang Bin Jusoh and Zaini Binti Embong for an irreplaceable sacrifice and continuous support from the beginning until now. Their love and concern are everything to me until finally, I managed to complete my research and thesis.

Lastly, I would like to convey my gratitude to all people who directly or indirectly give me a hand in the process of completing this thesis. It was one of the greatest experience learning and the fundamental knowledge during this particular period.

ABSTRAK

Logam campuran CoCr kebiasaannya digunakan untuk tujuan pembedahan kerana mempunyai sifat daya tahan daripada kakisan, kekuatan logam yang tinggi dan juga bersifat biokompabiliti. Proses pembentukan logam *(MIM)* adalah satu daripada proses metalurgi serbuk yang digunakan untuk menghasilkan suatu komponen yang rumit dan menepati kehendak bentuk. Kini, proses tersebut juga telah dijadikan sebagai salah satu alternatif bagi menghasilkan implan untuk tujuan pembedahan dan pergigian sesuai dengan kehendak pasaran perubatan yang tinggi. Proses pembentukan logam ini terhasil melalui teknik pencampuran logam dan komponen perikat, kemudian proses pengacuan injeksi antara logam dan komponen peirikat, seterusnya peleraian pengikat *(debinding)* dan pensiteran (*sintering)*. Fasa *Sintering* dan *debinding* adalah fasa yang terpenting di dalam proses pembentukan logam memandangkan kedua-dua fasa ini mampu mempengaruhi sifat-sifat produk akhir setelah proses sintering. Hal ini kerana, kegagalan memilih parameter yang sesuai sewaktu proses ini boleh menyebabkan kerosakan produk akhir lebih daripada 25%. Mudah rapuh, ruang rongga yang banyak semasa proses *debinding* akan menyebabkan produk akhir selepas proses *sintering* menjadi senang retak dan tidak berkualiti. Oleh itu, kajian ini bertujuan untuk menentukan parameter yang sesuai bagi fasa *solvent debinding*, kemudian mengkaji kesan parameter fasa *sintering* terhadap struktur mikro produk akhir dan juga kesan parameter sintering terhadap sifat fizikal, mekanikal dan sifat kakisan produk. Komponen pertama yang dihasilkan selepas proses pengacuan injeksi akan *didebound* melalui proses *solvent debinding* dalam larutan n-heptane pada suhu 50, 60 dan 70˚C selama 4 jam. Komponen kedua yang dihasilkan selepas proses *solvent debinding*, akan melalui proses sintering pada suhu 1250, 1300 dan 1350˚C selama 1 dan 3 jam dalam atmosfera argon. Setelah itu, komponen akhir yang terhasil selepas fasa sintering akan dikaji melalui kaedah ketumpatan, ujian ketegangan dan kekerasan mikro. Sifat kakisan ditentukan daripada kaedah elektrokimia. Manakala, kajian metallografi untuk permukaan patah dan pembentukan liang struktur mikro komponen dianalisis melalui microskop optic (OM) dan microskop pengimpasan electron (SEM). Hasil eksperimen telah menunjukkan, solvent debinding pada suhu 60˚C selama 4 jam ditentukan sebagai parameter yang sesuai untuk proses ini memandangkan peratus kehilangan perikat mencukupi iaitu 76.9%. Manakala, komponen yang disinter pada 1350˚C-3jam menunjukkan ketumpatan tertinggi (92.5%), kekerasan mikro (308.1 HV), kekuatan tegangan (728.5 MPa) dan kadar kakisan yang bagus, 2.16E-06 mm / tahun dibandingkan dengan sintered pada 1250˚C-jam, 1300˚C-jam, 1350˚C-1jam, 1250˚C-3jam dan 1350˚C-3jam. Apabila suhu meningkat, dan masa dipanjangkan untuk proses sintering, kekuatan tegangan dan kekerasan meningkat secara beransur-ansur. Manakala, struktur mikro komponen sintered menunjukkan bahawa ikatan morfologi antara partikel serbuk menjadi lebih padat apabila masa yang diperuntukkan sesuai iaitu 3jam dan suhu sintering yang tinggi pada 1350˚C. Tambahan pula, pengecilan ruang rongga di dalam struktur mikro komponen juga telah membantu menguatkan sifat mekanikal logam komponen CoCrMo. Kesimpulannya, suhu dan masa sewaktu proses sintering adalah parameter yang kritikal untuk kepadatan ruang rongga dan meningkatkan sifat-sifat mekanikal dan sifat-sifat kakisan kompak sinteran.

ABSTRACT

The CoCr-based alloy is used for surgical inserts due to their superior corrosion resistance, high strength and good in biocompatibility. Metal injection moulding (MIM) is one of the powder metallurgy process used for the fabrication of complex and near net shape assembly of high-performance materials. Currently, this method has also been used as an optional way to fabricate implants in surgery and dentistry befitting for economical mass production. MIM process involves mixing, injection moulding, debinding and sintering. Sintering and debinding is the main stage in the injection moulding process that could affect the properties of the sintered part. In some instances, no optimised parameters were apply to both process may cause defective for final products formed up to 25 %. Brittle, porous structure of brown compacts after debinding caused sintered compact after sintering process become easily cracks and not quality compacts were formed. This research interests in the persistence of optimum parameter for solvent debinding process, the effects of sintering parameters on microstructure of sintered compact and effect of sintering parameters on physical, mechanical properties and corrosion behaviours of sintered compacts. The CoCrMo alloy compacts were solvent debound in n-heptane solution at 50, 60 and 70˚C for 4 hours soaking time. The brown compacts produced was continued to a sintering process at temperature 1250, 1300 and 1350˚C for 1 and 3 hours in an argon atmosphere respectively. The tensile and microhardness testing determined the mechanical properties of sintered CoCrMo alloy compacts. The corrosion properties were characterised from electrochemical method experiments. Metallographic studies of fractures surface and pore formation were observed by optical microscopy (OM) and scanning electron microscope (SEM). The results of the investigation indicate that solvent debinding at 60 ˚C for 4 hours was found to be optimum parameters for solvent debinding due to adequate mass loss of paraffin wax which reported at 76.9 %. While compacts sintered at 1350˚C-3h showed the highest density (92.5 %), hardness (308.1 Hv) and tensile strength (728.5 MPa) and desirable corrosion rate of 2.16E-06 mm/year compared to the sintered conditions at 1250˚C-1h, 1300˚C-1h, 1350˚C-1h, 1250˚C-3h and 1350˚C-3h. As the temperature increased, and holding time was prolonged to 3 hours, the tensile strength and the hardness increased gradually. While, from the morphology of sintered compacts, it shows that the morphological bonding between powder particles become denser with longer sintering time, 3 h highest sintering temperature of 1350 ˚C. Moreover, the decrease in porosity amount was shown to improve the mechanical properties of CoCrMo alloy sintered compacts. It can be concluded that the sintering temperature and time is a critical parameter for reducing the amount of porosity and improving mechanical properties of sintered compacts.

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LIST OF SYMBOLS

<	Less than
>	More than
-	Subtraction
+	Addition
/	Division
=	Equal
x	Multiplication
( )	Calculate expression inside first
d_1	Average dimension of green compact
d_2	Average dimension of sintered compact
P	Applied load
A_0	Specimen's original cross sectional area
σ	Stress
δ	Change in the specimen's gage length
L_0	Original gage length
ε	Strain
D10	The range of particle size diameter at 10%
D50	The range of particle size diameter at 50%
D90	The range of particle size diameter at 90 %
µ	Micro
±	Both plus and minus operations

LIST OF ABBREVIATIONS

ASTM	American society for testing materials
At. %	Percentage in atomic
C	Carbon
Ca	Calcium
Cl	Chloride
CMC	Ceramic matrix composite
DSC	Differential scanning calorimetric
EDS	Energy dispersive x-ray
H	Hydrogen
H_2O	Distilled water
HCl	Hydrochloric acid
HNO_3	Nitric acid
CH_3COOH	Acetic acid
K	Potassium
MIM	Metal Injection Moulding
Mg	Magnesium
NiTi	Nickel-titanium
MPIF	Metal powder industries federation
N	Nitrogen
Na	Sodium
O	Oxygen
OM	Optical microscope
PW	Paraffin wax
PP	Polypropylene
SA	Stearic acid
SEM	Scanning electron microscope
CoCr	Cobalt chromium-based alloy
CoCrMo	Cobalt chromium molybdenum alloy
TGA	Thermogravimetric analysis
Wt.%	Percentage in weight
Vol.%	Percentage in volume

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